

# FlatLand → MicroLand → NanoLand

## **Metrology and Standards: Challenges in Micro/Nano Materials and Systems**

NIST USMS Workshop  
September 22, 2005

Robert Scace, Klaros Corporation  
Winthrop Baylies, BayTech Group



# FlatLand → MicroLand → NanoLand



## Outline

- Micro Standards Topics
  - SEMI MEMS
    - Wafer Bond Targets
    - Bond Integrity Tests
    - Bond Strength Tests
    - Terminology
    - Fluidics Design Guide
  - ASTM Film Stress Tests
- Nano Standards Topics
  - SEMI Nanoparticles
  - IEEE Carbon Nanotubes (CNT)
- Other NANO Activities
  - ISO TC229 on Nanotechnologies
  - ANSI NSP (Nanotechnology Standards Panel)
  - ASTM Committee E56 on Nanotechnology

# FlatLand → MicroLand → NanoLand



- FlatLand
  - Edwin A. Abbott's classic tale of interdimensional experience: <http://www.alcyone.com/max/lit/flatland/>
  - What we think we see based on CMOS
- MicroLand
  - What we think we know
- NanoLand
  - What we think we imagine



# MicroLand

22 September 2005

NIST ASMS Workshop  
Pittsburgh

4

# SEMI MEMS Wafer-Wafer Bonding Activity

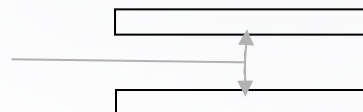


- *Doc 3950 – Guide to Specifying Wafer-Wafer Bond Alignment Targets*
  - March 05: Initial Ballot
  - August 05: International Ballot
- **Wafer Bond Tests**
  - Integrity (Voids)
    - Surveyed Test Equipment Users, Suppliers
    - Interpretation underway
    - Guide for Specifying Equipment – Draft 1/October
  - Strength
    - Micro-Chevron test
    - Work starting Oct 2005

# Wafer Alignment Processes

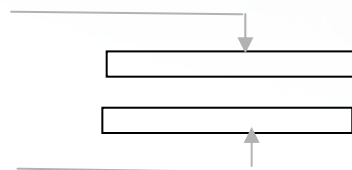


- ISA – Inner Substrate Alignment



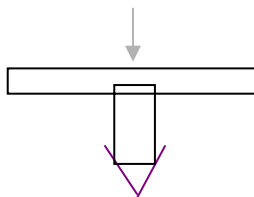
Balloted July05

- BSA – Back Substrate Alignment



Balloted July05

- TSA – Top Substrate Alignment

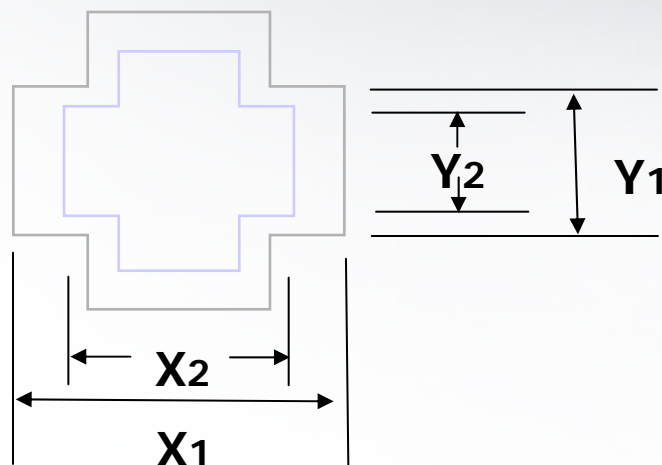


Not balloted  
Future Consideration

# Suggested Alignment Targets



## Geometry



### Example

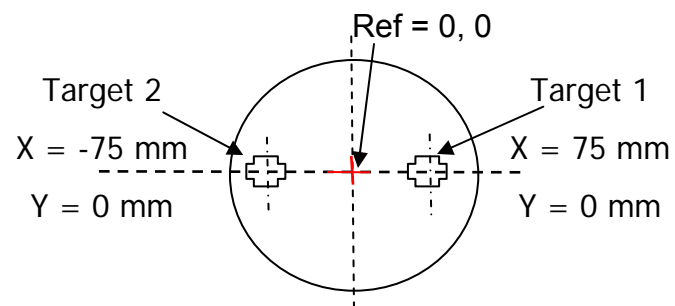
$$X_1 = 50 \mu\text{m} \pm 5 \mu\text{m}$$

$$X_2 = 40 \mu\text{m} \pm 4 \mu\text{m}$$

$$Y_1 = 50 \mu\text{m} \pm 5 \mu\text{m}$$

$$Y_2 = 40 \mu\text{m} \pm 4 \mu\text{m}$$

## Location



# MEMS Materials Characterization TF

## Wafer Bond Tests



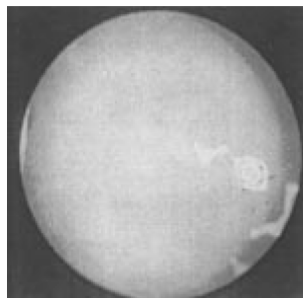
- Bond Integrity Survey
  - Distributed in April to Metrology Tool Makers
- Returns – Metrology Tool Suppliers/Users
  - 5 IR Transmission, 4 Ultrasound, 1 X-Ray Topography
  - Summary Comparison / Analysis
    - In Progress
  - Draft “Guide for Selecting Wafer Bond Integrity Measurement Systems”
    - Draft 1 – October, Portland OR discussion



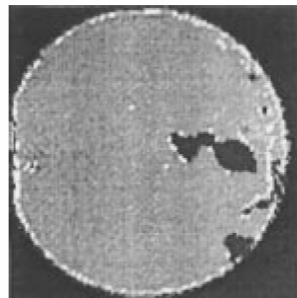
# Bond Integrity Inspection Issues



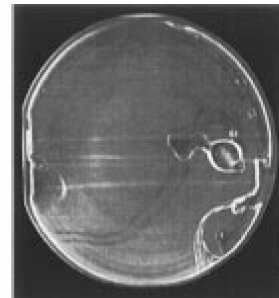
- Nondestructive evaluation of bond interface is critical for quality control in wafer-wafer bonding processes
- Several methods have been used:



IR transmission



ultrasound



x-ray topography

Images from M. Schmidt, Proc. IEEE 86:1575 (1998)

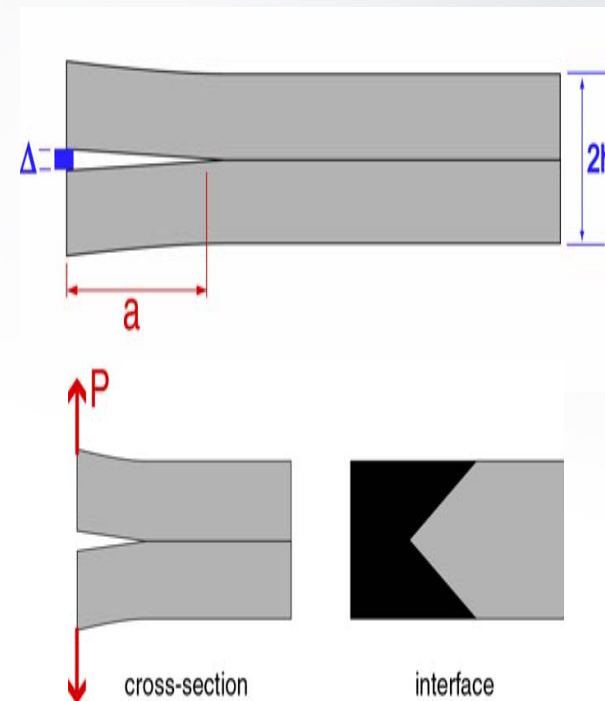
- Differing gap and spatial resolutions, process compatibility, suitability for certain materials systems, etc.
- Qualitative understanding of differences between methods exists, but **quantitative** limits are unclear
- GOAL: Develop table of inspection methods and attributes that facilitates user selection of techniques

# MEMS Materials Characterization TF

## Bond Strength Test

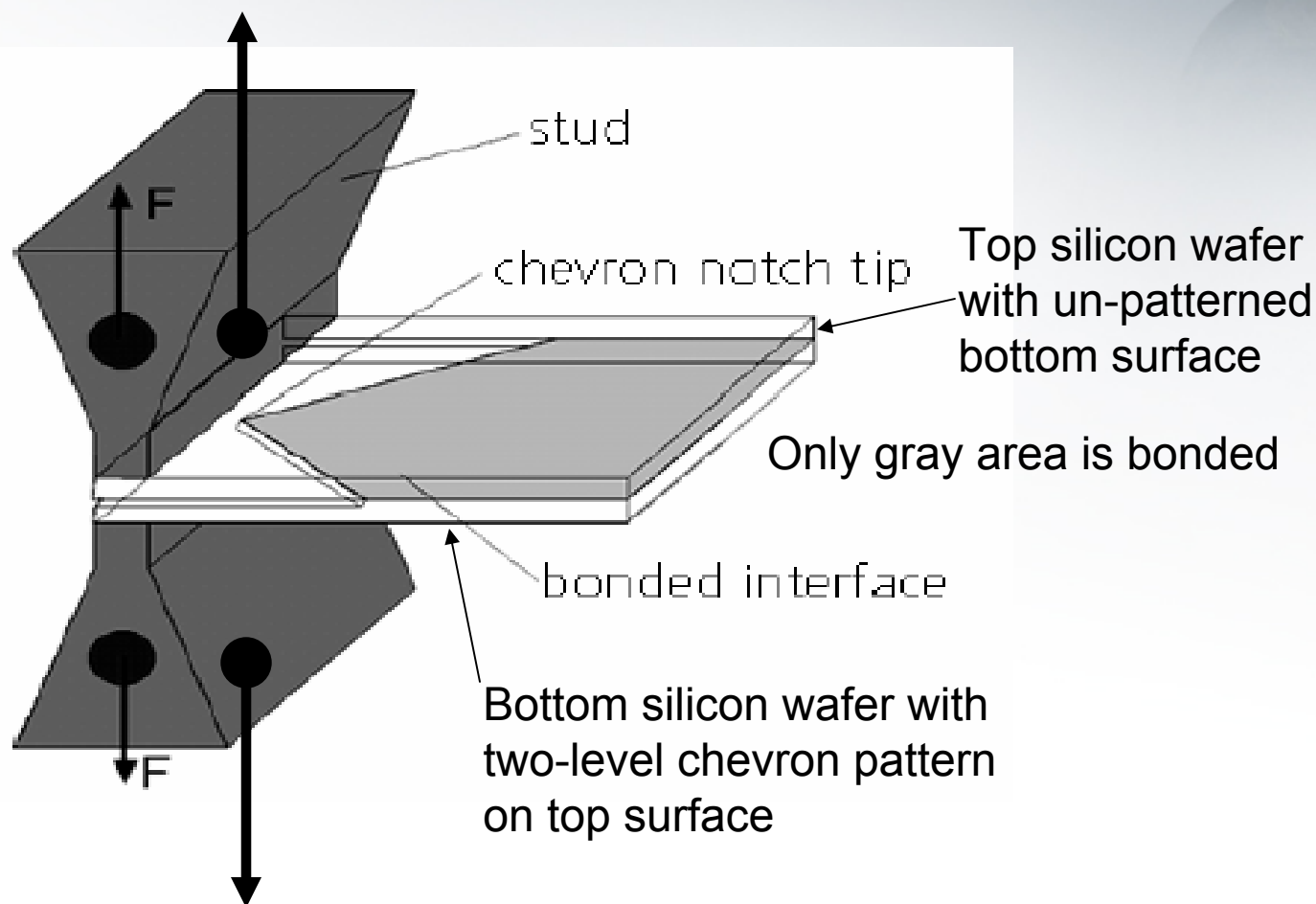


- Interface Strength Affected By
  - Wafer Pre-treatment
  - Bonding Conditions
- Test Types
  - Crack Propagation
    - Large physical sample size, Measurement Uncertainty
  - Chevron
    - Smaller physical sample size, Improved Measurements
  - Micro-Chevron
    - Smallest samples, High spatial resolution/precision
    - Spatial Strength Distributions

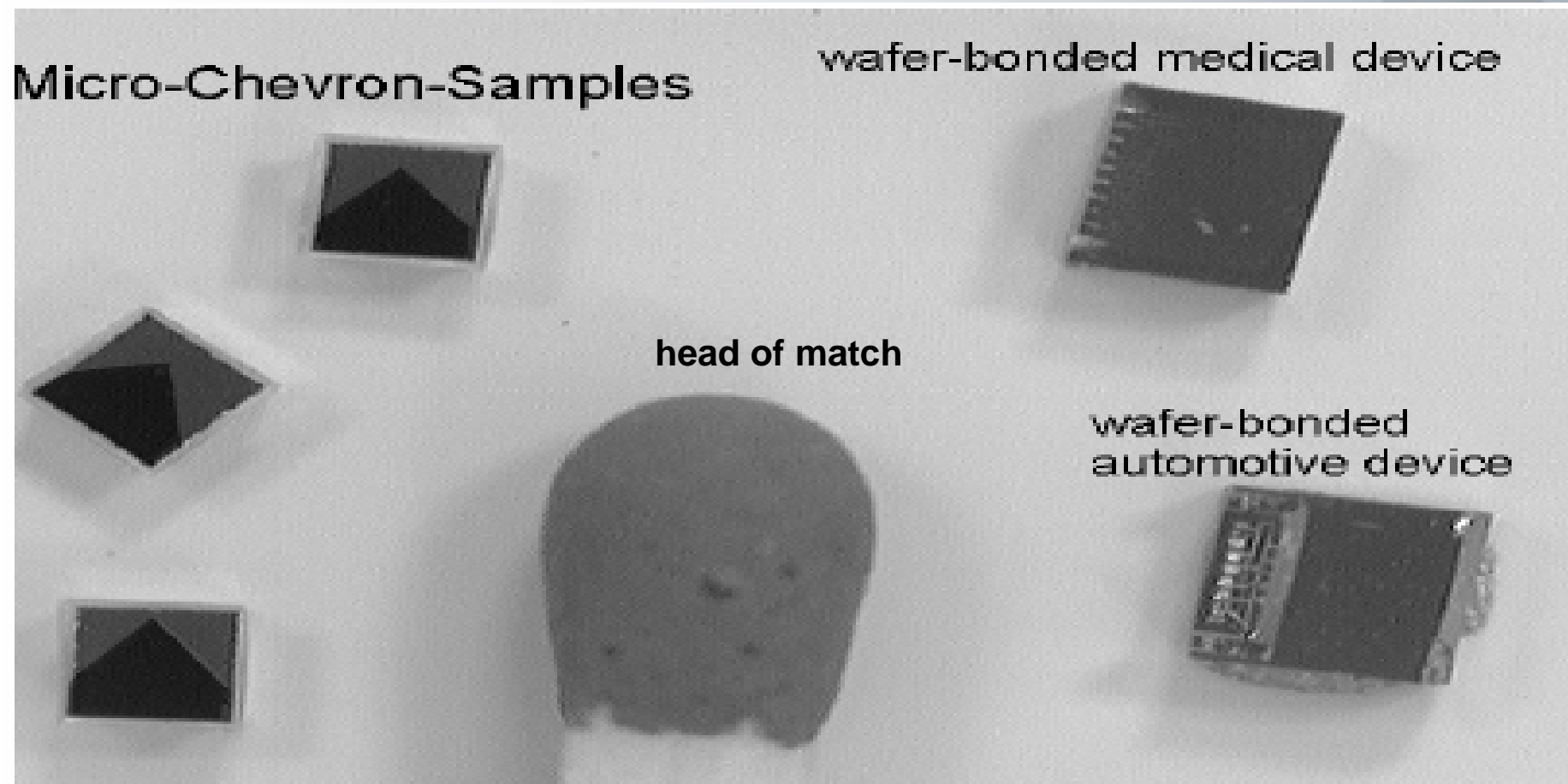


**Provides a measure of the interface toughness – units of energy/area –  $\text{J/m}^2$**

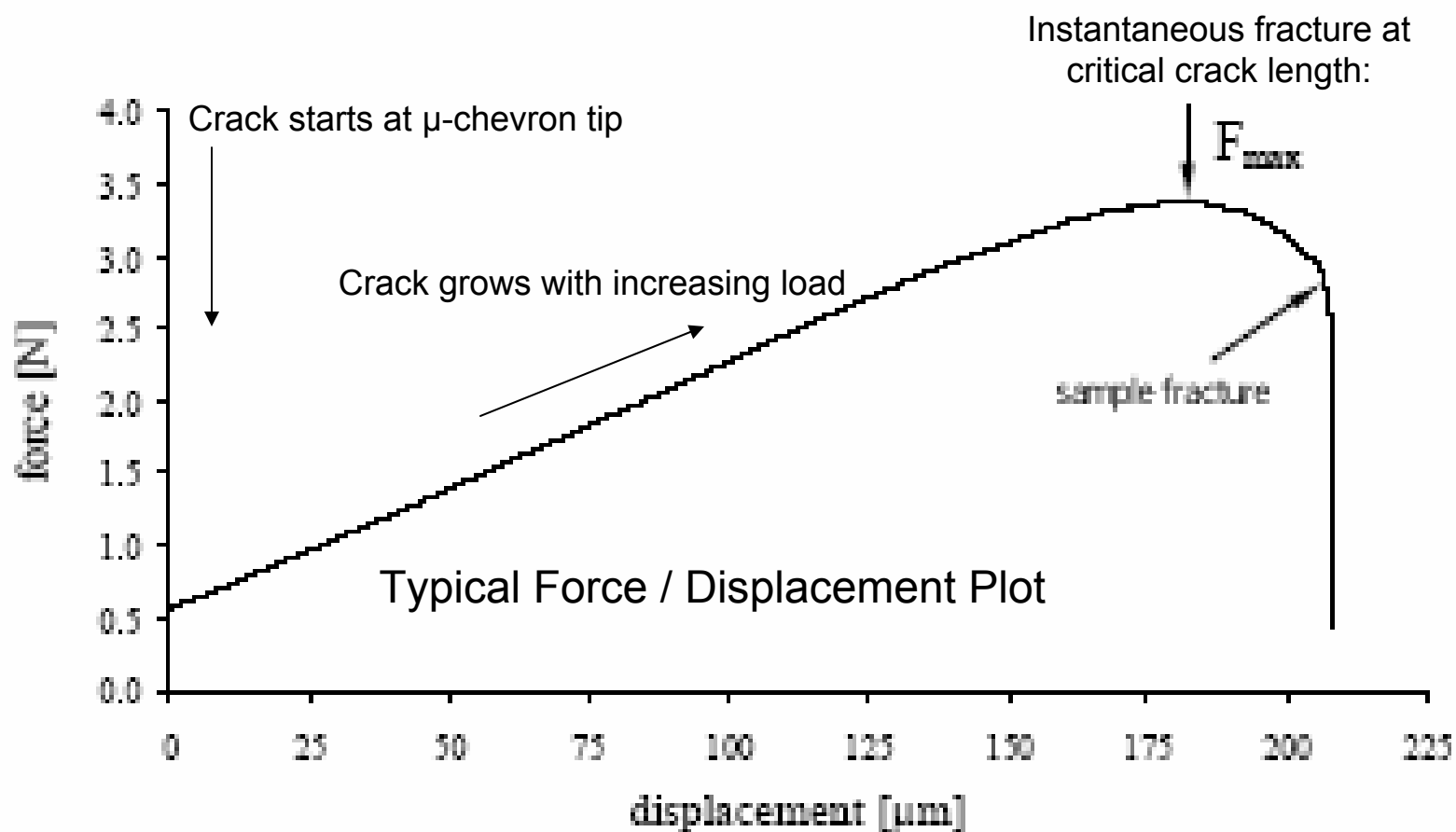
# Micro-Chevron Testing



# Micro-Chevron Comparison



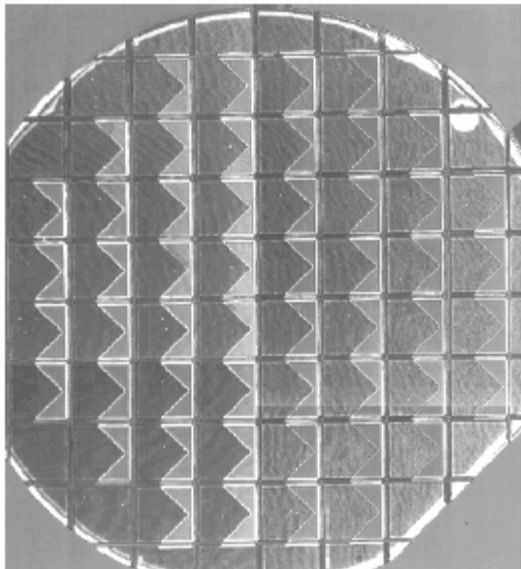
# Micro-Chevron Test Example



# Micro-Chevron Experiment Strength vs. Position

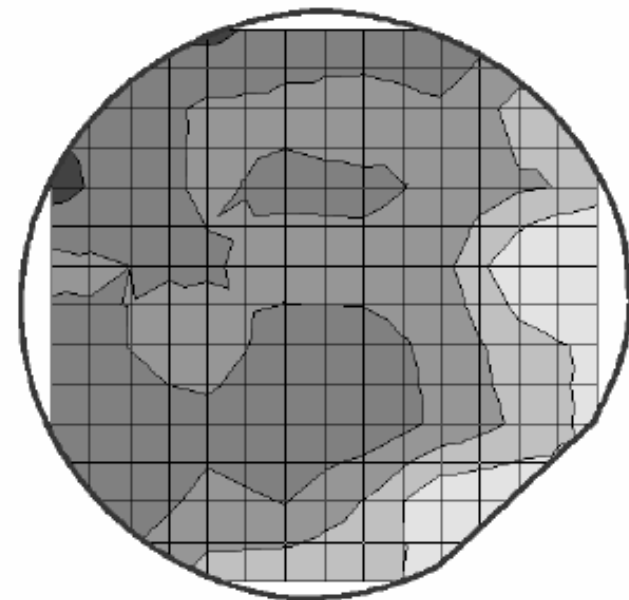


Sample Array Interface  
Si-Si Wafer Pair #1



Source: Bagdahn *et al*; Fraunhofer  
Institute for Mechanics of Materials

$\Delta$  Strength Distribution  
Si-Si Wafer Pair #1

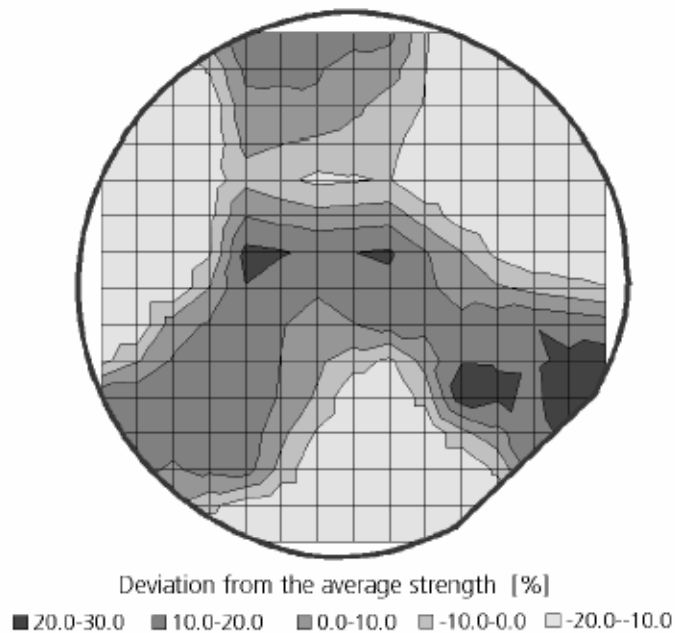


Deviation from the average strength [%]  
□ -20...-12   ■ -12...-4   ■ -4...+4   ■ +4...+12   ■ +12...+20

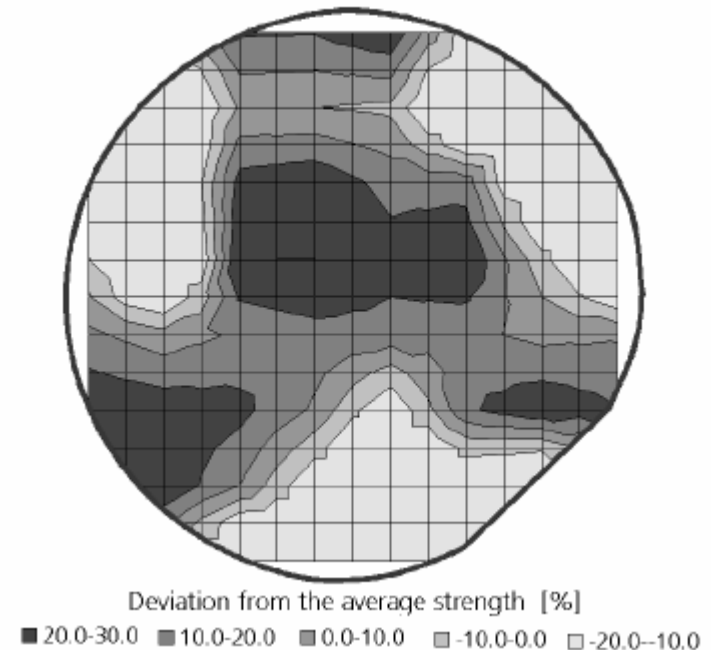
# Micro-Chevron Experiment Strength vs. Position 2



Δ Strength Distribution  
Si-Glass Wafer Pair #1



Δ Strength Distribution  
Si-Glass Wafer Pair #2



Source: Bagdahn *et al*; Fraunhofer Institute for Mechanics of Materials



# **MEMS Fluidics Task Force Design Guide**

Guide for Standard Performance, Practices, and  
Assembly for Ultra High Purity Microscale Fluidic  
Systems for Use in Scalable Process Environments

- Outlines necessary topics; includes source references
- Design considerations; materials issues; connections; subsystems; interfaces
- Published as Preliminary Standard 7/05
- Development continues

Note: Project defined and led by a major semiconductor manufacturing equipment manufacturer



# MEMS Terminology Task Force



- Global SEMI collection effort
  - Initial draft of nearly 150 definitions, many duplicates
  - Weeded, edited, re-formatted
  - Reviewed for technical issues
  - Approved as SEMI Preliminary Standard 7/05
    - Contains 74 terms
  - Revisions and additions already under way

# ASTM Film Stress Test Methods



## **Basic tool is non-contact optical interferometry**

- E 2244: in-plane length of thin films
- E 2245: residual strain in thin films
- E 2246: strain gradient in thin films
  - NIST round-robin tests of these methods aided by SEMI cooperation
  - ASTM terminology from these standards is included (by permission) in SEMI Preliminary Standard
- E 2444 Terminology Relating to Measurements Taken on Thin, Reflecting Films



# NanoLand

# SEMI Nanoparticles in Slurry for CMP (Chem-Mechanical Polishing)



- 2Q05 Survey – Slurry Users, Material Suppliers
  - Goal: Develop *Guide for Characterizing Nanoparticles for Use in CMP Slurry*
  - Review Draft in Portland Oregon October 10
- Nanoparticle:  $\leq 100$  nm equivalent diameter, after capillary membrane separation
- Manufacturing Processes:
  - Pyrogenic, TEOS Hydrolization, Fumed SiO<sub>2</sub>
  - Resultant Compositions: Al<sub>2</sub>O<sub>3</sub>, CeO<sub>2</sub>, SiO<sub>2</sub>
- Size distribution: 20 – 80 nm, typical

# SEMI Nanoparticles in CMP Slurry 2



Important Characteristics	2005	Future
<b>Surface</b>		
Smooth, Nearly Spherical	50%	60%
Highly Irregular	40%	10%
<b>Electrical</b>		
Electrochemical Surface Potential (when in slurry)	50%	40%
<b>Purity</b>		
High < 1000 ppb	10%	10%
Highest < 1 ppb		10%

# SEMI Nanoparticle Metrology



- In Use
  - SEM 60%
  - Others: Particle Size Analyzer, Particle Size Distribution, Abrasive Surface Charge

- Challenges

- SEM for  $< 100$  nm; non-dry condition
  - Fast Particle Surface Geometry
  - Chemical Property Analysis

	<i>2005</i>	<i>Future</i>
• Dispersion	X	X
• Hydrophilic	X	X
• Free Metal Pollution	X	
• Low Pollution		X

# IEEE Standards for Nanotechnology



- IEEE P1650<sup>TM</sup>, Standard Test Methods for Measurement of Electrical Properties of Carbon Nanotubes
  - Initial Sponsor Ballot closed 8/18/05
- IEEE P1670<sup>TM</sup>, Chemical Vapor Deposition Techniques for Nanotechnologies Including Measurements and Analysis to Control CVD Nanoscale Processes – under development
- IEEE Quality Study Group on Carbon Nanotubes
  - Project approval being sought

# ASTM Committee E56 on Nanotechnology



- Proposed a year ago; over 100 members to date
- More than 16 countries represented
  - Vice Chairman is from AIST
- Subcommittees:
  - Terminology and nomenclature – draft balloting now
  - Characterization – first draft test method balloting now
  - Environmental and Occupational Health and Safety
  - International Law and Intellectual Property
  - Liaison and International Cooperation
  - Standards of Care and Product Stewardship



# ISO TC229 on Nanotechnologies



- Proposed by UK 1/05
- Supported by ANSI and 24 other countries 4/05
- ISO authorized TC formation 7/05
  - UK has TC secretariat
  - Proposed subcommittees and probable secretariats:
    - Terminology and Nomenclature – UK
    - Metrology – Japan
    - Environmental and Occupational Health and Safety – US
- First meeting November 9-11 in London



# Questions?